

Chapter 13

Production of neutral spirits and preparation of gin and vodka

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Introduction

Neutral spirit is basically purified, odorless, tasteless, colorless ethanol (or ethyl alcohol -

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OH). It may be produced from almost any fermentation feedstock if suitable distillation and rectification techniques are used to remove the other chemical compounds, or congeners, produced with the ethanol in the fermentation process.

Neutral spirit is used in the production of beverages such as vodka, gin, cordials and cream liqueurs. It is also used for a wide range of other industrial manufacturing applications, where it is usually referred to as "high quality industrial alcohol". Purified synthetic alcohol produced from petrochemicals may also be used in industrial applications, being virtually indistinguishable from neutral spirit produced from fermentation feedstocks, but it is outside the scope of this chapter.

Feedstocks

Almost any fermentation feedstock may be used to produce neutral spirit, but certain feedstocks may produce greater concentrations of particular congeners that require additional equipment for removal. For instance, potatoes and grapes tend to produce much higher levels of methanol than are obtained from grain fermentations; and it may be necessary to use an additional distillation column, a demethylizer, to obtain a satisfactory product.

The choice of feedstock is largely dictated by economics and availability. Thus for part of the year a plant may use a low cost, seasonally available, perishable feedstock such as sugarcane juice, grape juice, or cheese whey. The rest of the year the plant will operate using more expensive but storable feedstocks such as grain, raisins or molasses.

Feedstocks for neutral spirit production must be consistently of good quality. They must be free of major bacterial infections in order to maintain fairly clean fermentations and avoid excessive variation in the amount and type of congeners to be removed in subsequent processing.

Fermentation

The preparation and fermentation of the various feedstocks is essentially the same as described in the chapters on the production of whisky, rum and fuel ethanol. However, because the objective is to produce lower concentrations of congeners (since no distinctive flavor is required), more emphasis may be placed on use of bactericides and antibiotics (e.g. chlorine dioxide and penicillin) to control bacterial contaminants in the fermenters.

The fermentation process yields a beer containing varying concentrations of ethanol depending on the feedstock used. For example, the ethanol concentration from fermentation of sulfite waste liquor (from the paper/pulp industry) may be only about 1% by volume. Cheese whey fermentations normally yield 2-5% ethanol; sugarcane juice fermentations may yield 6-8% ethanol and grain fermentations may yield 10-12% ethanol.

Distillation and rectification

Beer distillation

Conventional US process

The first stage in the distillation process is separation of the ethanol and most of the congeners from the bulk of the liquid and solids in the fermented beer. This is normally carried out in a beer still similar to that shown in Figure 1.

The beer still usually consists of two columns.

The first column is the beer stripper and the second is the concentrator. In some instances the concentrator may be stacked on top of the beer stripper to make a single, tall column. The beer stripper may contain about 20-25 sieve trays, or it may have other contact devices such as disc-and-donut baffle trays.

The beer is fed into the beer stripper near the top, while live steam is introduced at the bottom of the column. (A reboiler may be used at the bottom of the column for indirect heating, but requires some provision to avoid scaling from the beer solids.) The ethanol and most of the congeners are stripped from the beer by the steam (as described in the chapter on distillation) and they pass in vapor form to the concentrator. This column may have about 30-40 trays with sieve holes, bubble caps, or tunnel caps. For convenience and clarity, the concentrator

in Figure 1 is shown as having overhead condensers with gravity reflux of condensate back to the column. In many instances, particularly in recent installations, the condensers may be situated near ground level with the reflux being pumped back up to the top of the concentrator.

The ethanol rises through the concentrator and may be concentrated to about 95

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GL. It is drawn off from a valve located a few trays below the top of the column and sent to a surge tank for temporary storage as an intermediate product.

If the conditions in the concentrator can be held in a steady state, various congeners will accumulate at different levels in the column. Isoamyl alcohol, which is usually the principle higher alcohol or fusel oil congener in the beer, tends to accumulate where ethanol concentration is about 65

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GL (This may be about two or three trays above the fusel draw tray.) These compounds may be drawn off in a small purge flow and sent to storage for subsequent reprocessing to recover the ethanol in the flow. The concentration peaks of the congeners in a typical beer still are shown in Figure 2. (For clarity and simplicity the beer stripper and concentrator are shown as a single column.) It will be seen that if the peak heights are not controlled below certain limits, the congeners will extend up the column and be present in significant amounts in the 95

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GL intermediate product discharge line to maintain a steady proof of about 65

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GL intermediate product. To avoid this, European beer stills usually include a heads removal section on top of the beer stripper as shown in Figure 3. In this system (developed by Emile Barbet (1922) of France, described by Mariller (1943) and copied extensively in Brazil and other countries) the beer passes through a pre-stripping section of four or more trays where the more volatile components, including the

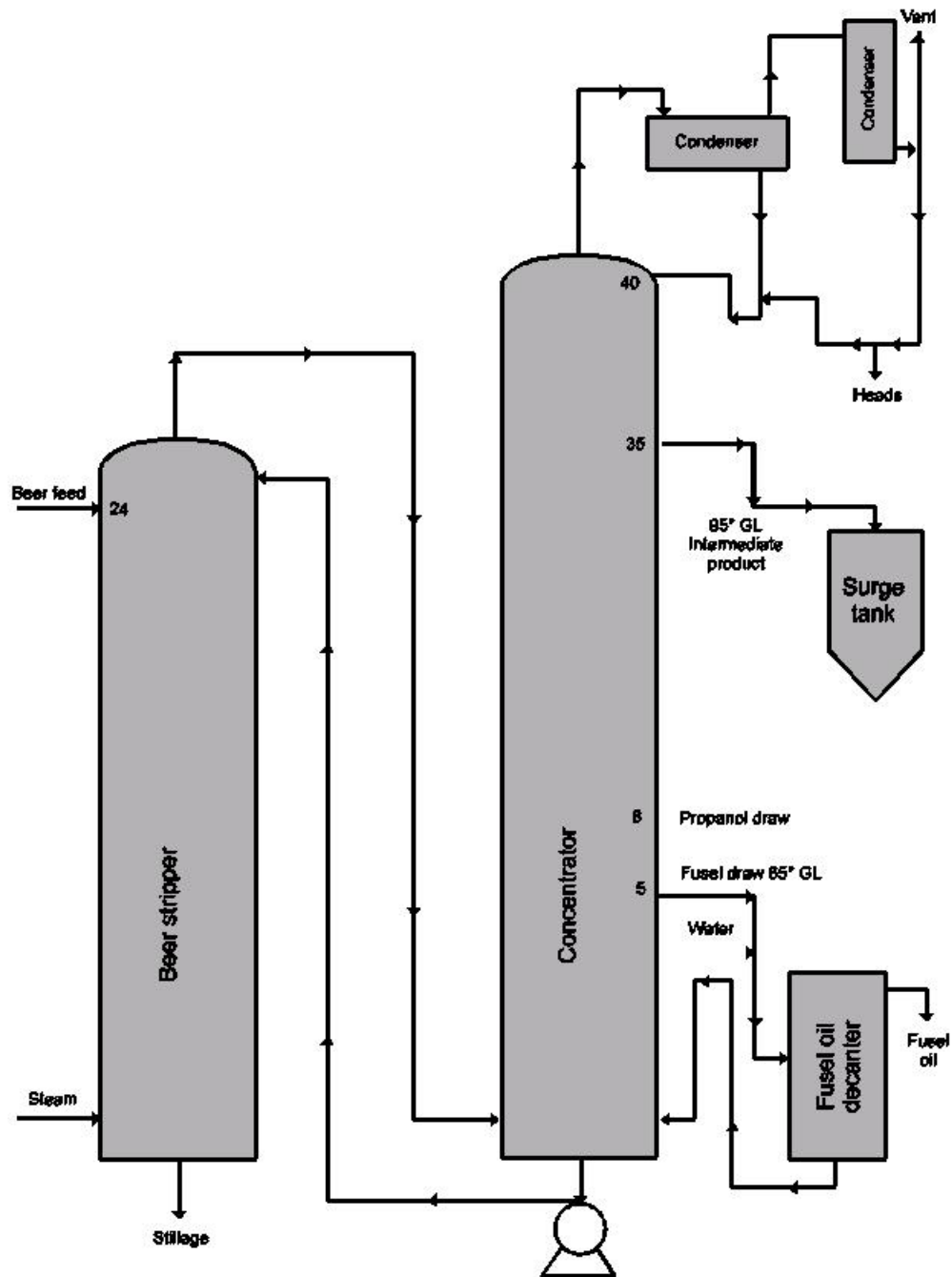


Figure 1. Conventional beer still used in neutral spirit production.

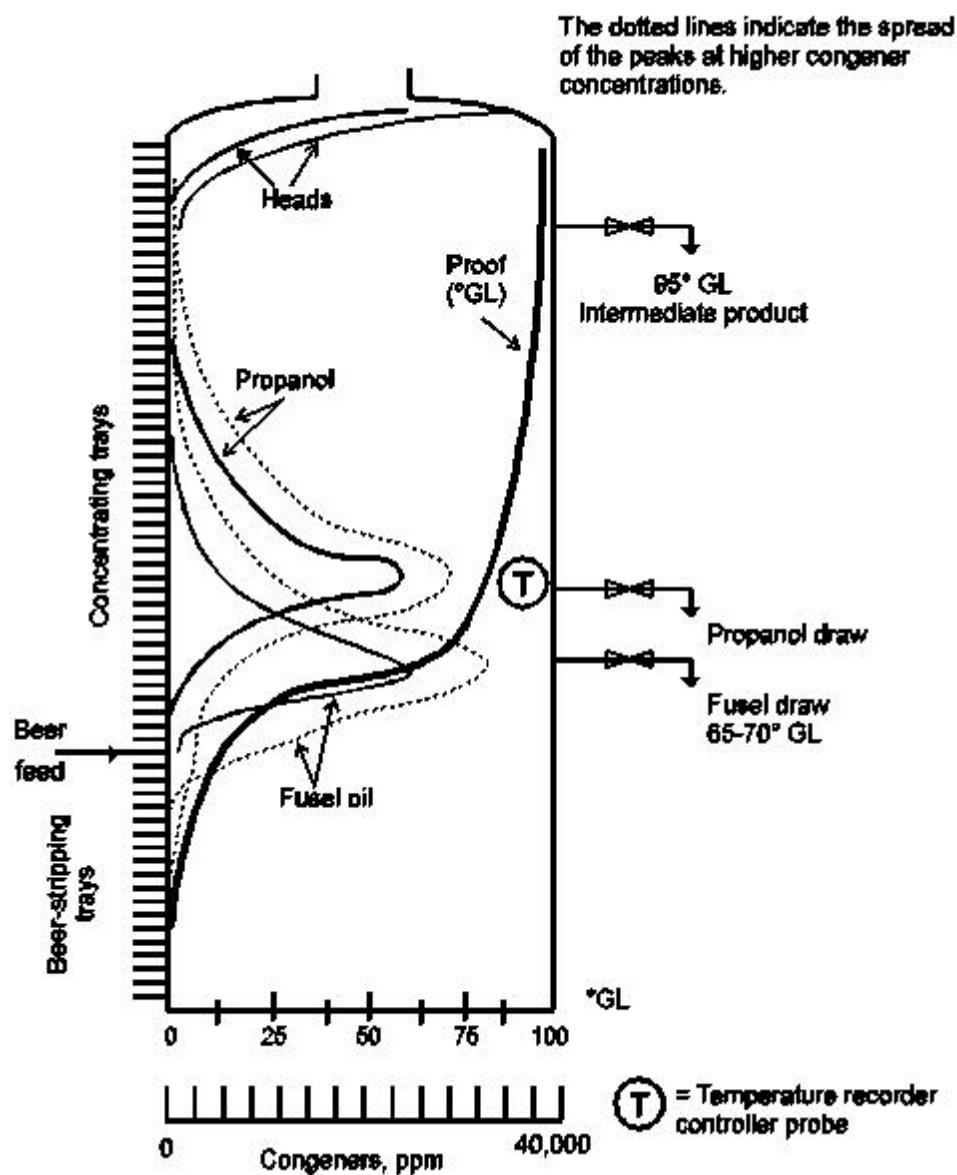


Figure 2. Congener peaks in a beer still with alcohol proof curve superimposed.

heads, are flashed off. The vapor rises into a heads concentrating section of six or more trays; and the heads may be drawn off as a purge stream from the reflux from the condenser.

Meanwhile, the main bulk of ethanol and other congeners passes via the vapor line at the base of the pre-stripping section to the concentrator. Splitting of the vapor flows to the heads concentrating section and the concentrator is regulated by a manually controlled valve located either on a vapor line between the pre-stripper and the heads concentrator or in the vapor line to the condenser.

The Barbet system not only removes much of the heads before the concentrator, it has an added advantage in that it permits removal of carbon dioxide in the beer feed to prevent it taking up space in the concentrator and reducing its capacity. The Barbet system also allows other volatile impurities such as sulfur compounds from molasses feedstock to be vented or removed in the heads.

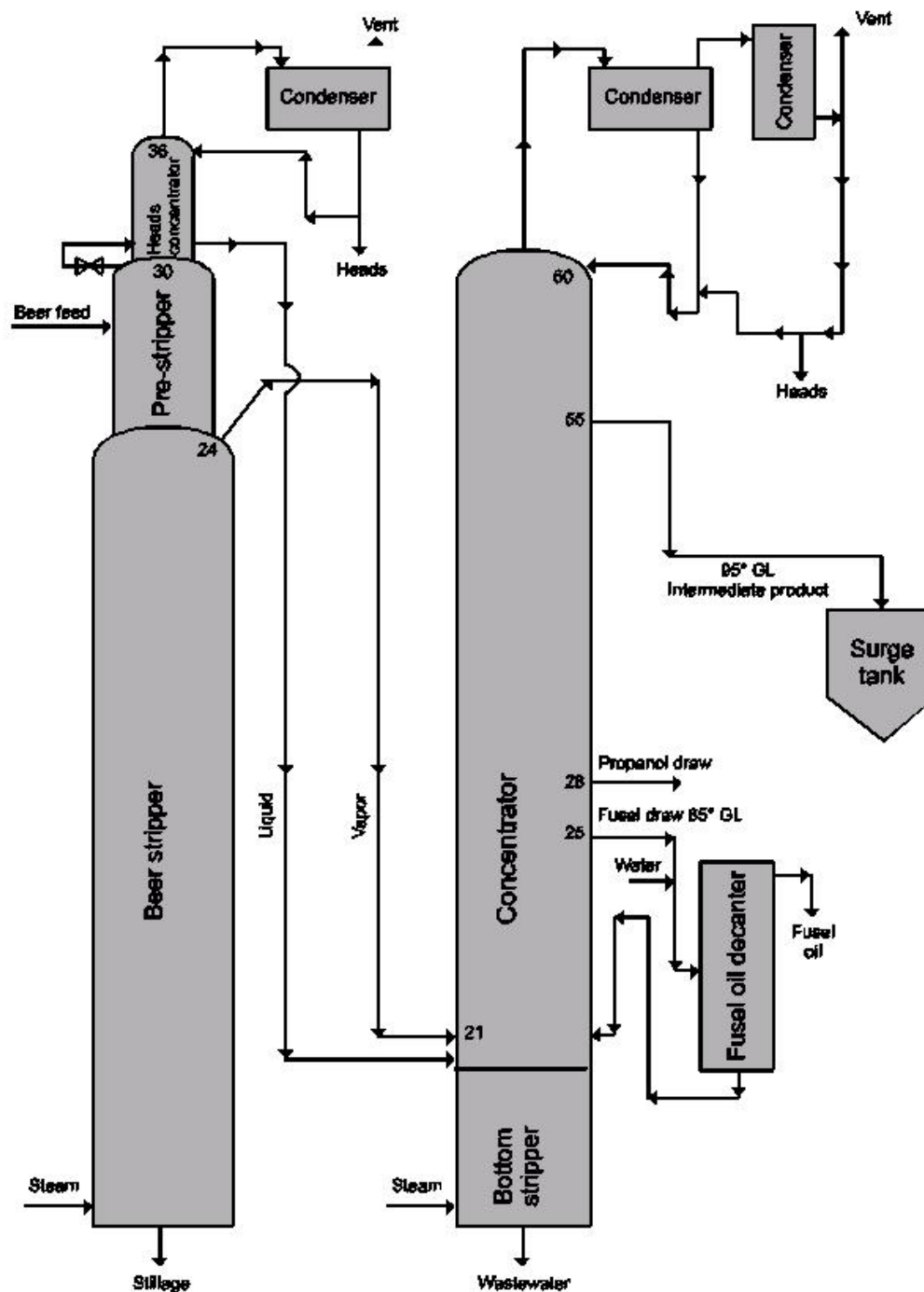


Figure 3. Modified Barbet still used in neutral spirit production.

Some Barbet systems also use the heads concentrating section on top of the beer stripper for concentrating heads piped back from the ethanol concentrator and from the rectification system. Conventional US systems normally have a separate heads concentrating column. Another variation common on modified Barbet beer still systems is a bottoms stripping section below the concentrator (as shown in Figure 3). This might be just a downward continuation of the concentrator with about 20 stripping trays, or it may be a separate column, usually of a smaller diameter. Having a bottoms stripper on the base of the concentrator means there is no need for a reflux line from the concentrator to the beer stripper. This avoids any ethanol return to the beer feed tray to cause scaling on the beer feed tray and reduce its capacity. This is of particular significance when using blackstrap molasses as the feedstock as it contains considerable quantities of calcium

compounds that contribute to scaling. Also to avoid scale there may be a line to take the reflux from the bottom tray of the heads concentrating section down to the bottoms stripper below the concentrator.

Another variation in beer distillation is to have a greatly reduced number of trays in the ethanol concentrator. Some beer stills may have only six trays in a concentrator located on top of the beer stripper. This means that the intermediate product proof will be much lower, at about 70

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GL. A beer still of this type is considerably less expensive in capital costs of fabrication. However, there is very little saving in operating costs, as there is not much reduction in steam usage because the energy input required to adequately strip the ethanol from the beer is also sufficient to operate the ethanol concentrator to near the 95

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GL point. A reduced number of ethanol-concentrating trays also means there is no provision for removal of fusel oil and other higher alcohols, so the only way they can be removed is in the rectification process. Reducing the number of ethanol-concentrating trays also reduces the capacity for concentrating the heads at the top of the column. Thus more heads will tend to come out in the intermediate product.

Rectification

Extractive distillation

Depending on the type and efficiency of the beer distillation system, the intermediate product will have some noticeable odor and will contain varying amounts of higher alcohols and heads that must be removed in the rectification process to yield a truly neutral spirit. However the congeners do not separate very well from the ethanol in the 95

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GL intermediate product by normal fractional distillation. For this reason it is necessary to employ an extractive distillation technique using water.

It should be noted that the efficiency of separation of the congeners can be increased greatly if the intermediate product is dehydrated to 100

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GL. Young (1988) proposed in a US patent that the intermediate product should be dehydrated and then subjected to two fractional distillation steps to remove the congeners that are both more and less volatile than ethanol. With the advent of molecular sieve ethanol dehydrators, this process could possibly be more cost effective than extractive distillation of the 94

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GL intermediate product.

The extractive distillation process relies on the fact that some of the congeners with higher boiling points that are normally less volatile become more volatile than ethanol in the presence of water. Thus, in a rectification system (as shown in Figure 4) the intermediate product is diluted in an extractive distillation column to remove the congeners as a heads stream. The diluted ethanol is then reconcentrated in the rectifying column.

The extractive distillation column may have about 40 trays. The 95

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GL feed from the intermediate product surge tank enters the column at about tray 30; and hot water is added on the top tray while live steam is injected at the base. By regulating the feed rate, dilution water and steam flow, most of the congeners will tend to go up the column and accumulate in the reflux loop to be drawn off in a heads purge. The optimum conditions will vary for the different

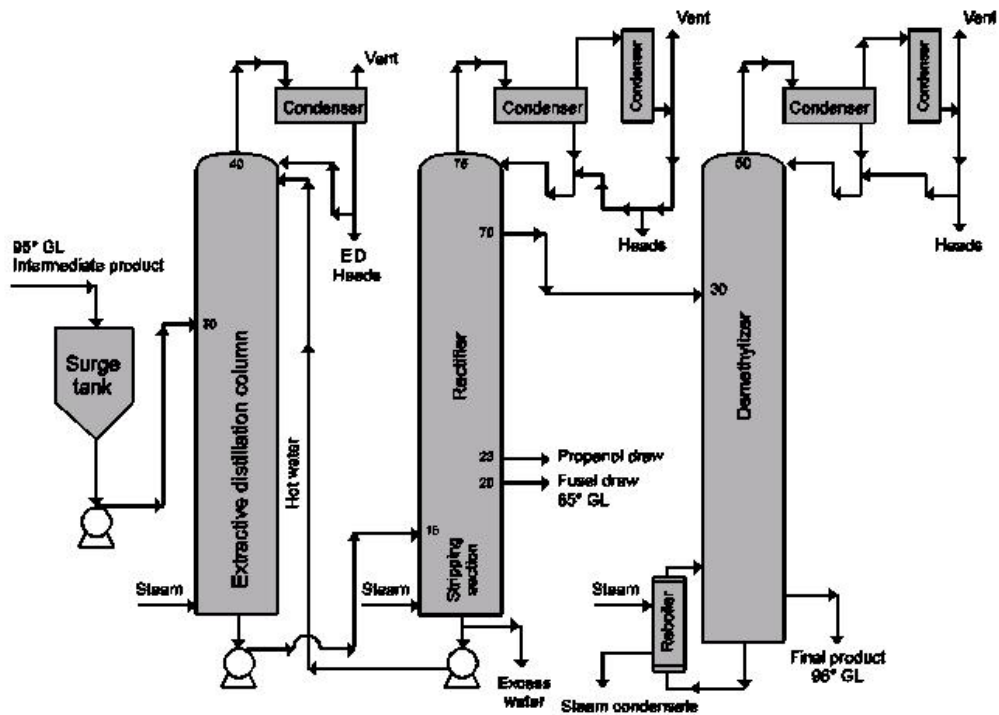


Figure 4. Three column rectification system used in neutral spirit production

congeners; so some compromise will be necessary depending on the mix of congeners present.

The theory of extractive distillation and its practical implementation in ethanol rectification has been covered very extensively by Chambers (1951, 1953, 1959). Two graphs derived from the Chambers publications are presented in Figures 5 and 6. Figure 5 indicates how the optimum conditions for removal of isoamyl alcohol and n-propanol may be established by comparing the proof in the "pinch zone" of the column (around tray 15) with the proof at the base of the extractive distillation column. The graph shows separate extraction limit lines for n-propanol and isoamyl alcohol; and it will be noted that less water and more steam are required to remove the latter than the former. The product obtained with conditions generally in the area between the n-propanol and isoamyl extraction limit lines is described by Chambers as "bland quality", while the product obtained wholly within the n-propanol extraction limit line is described as "vodka quality". Figure 6 shows how the efficiency of fusel oil removal is affected by the steam usage in the extractive distillation column and the proof at the base of the column. It will be noted that the optimum working area is where the base proof is relatively low and the steam usage is relatively high.

Final rectification

The dilute ethanol stream emerging from the base of the extractive distillation column will contain

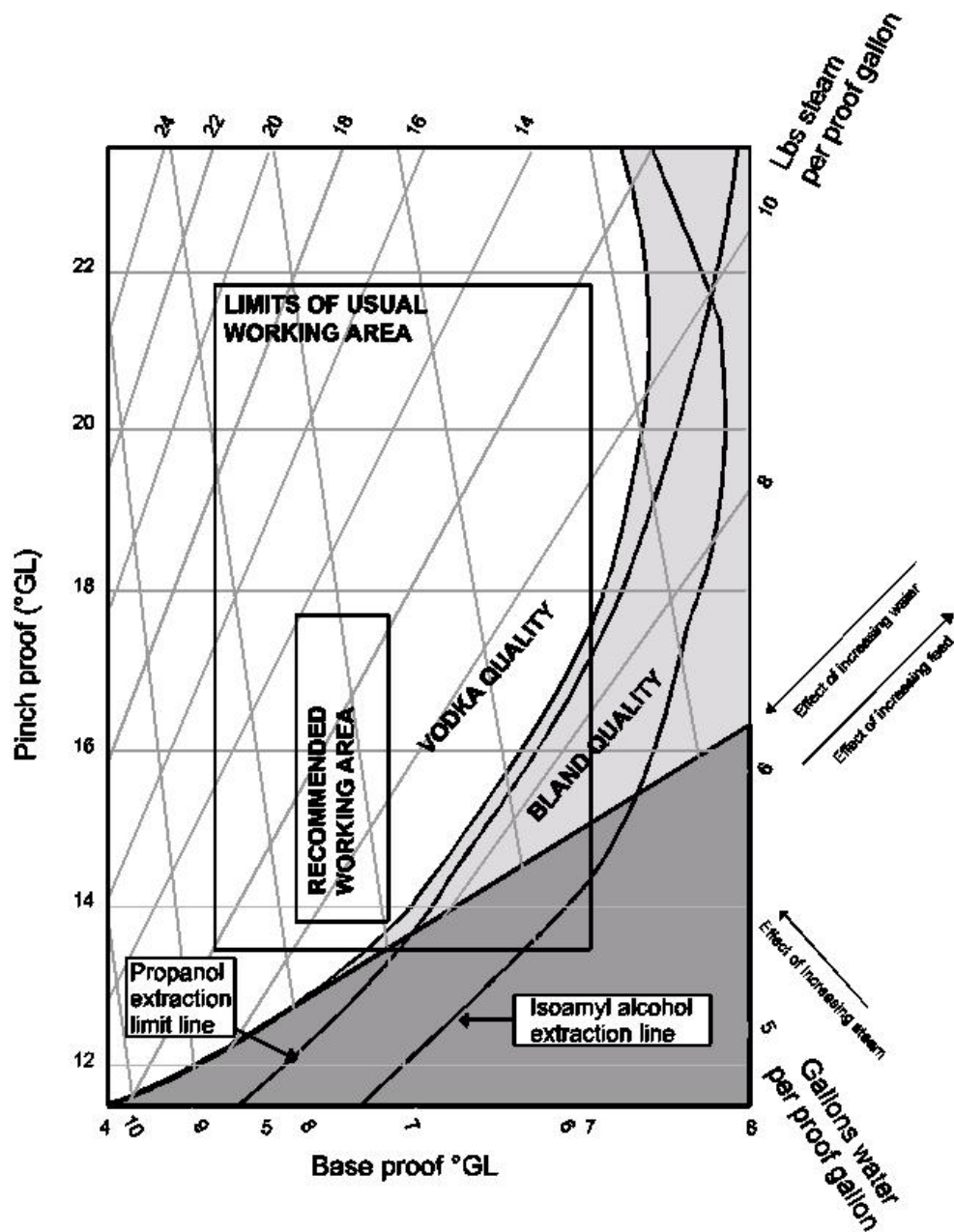


Figure 5. Chart for establishing optimum conditions for removal of propanol and isoamyl alcohol from an extractive distillation column.

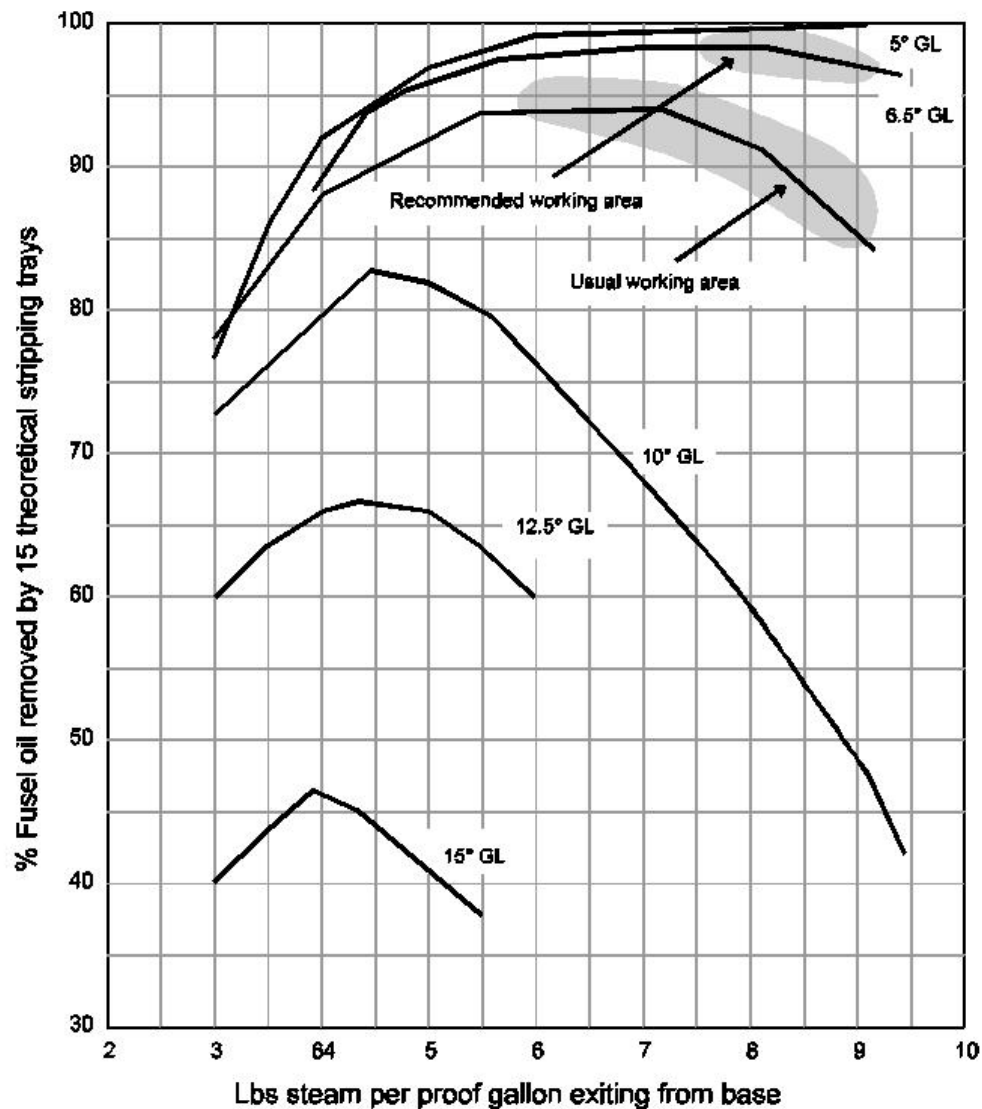


Figure 6. How the efficiency of extractive distillation varies with the base proof and steam usage.

the congeners not removed in the column heads. The congeners will include methanol, diacetyl, some aldehydes and relatively low concentrations of isoamyl alcohol, isobutanol and n-propanol. This stream is reconcentrated in a rectifying column (or rectifier) that usually has about 75 trays.

The feed stream from the extractive distillation column enters the rectifier at about tray 15. Live steam is injected at the bottom of the column, and the lower trays serve as a stripper to separate the water from the ethanol and congeners. The water emerges from the base of the rectifier; and part of the flow is recycled to supply the hot water requirements at the top of the column.

The ethanol and congeners travel up the rectifier and concentrate in much the same fashion as in the beer still concentrator. Thus the residual isoamyl alcohol accumulates at about 65

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normally near tray 20, where it is drawn off as a purge stream to be sent back to the beer still.

The traces of other high alcohols, mainly isobutanol and n-propanol, accumulate at about 80

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GL around tray 23 and are similarly recycled to the beer still.

The rectifier product is drawn off at about 96.1

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GL, usually about five trays from the top of the column (i.e. at plate 70), to leave some trays for heads concentration. The heads are drawn off as a purge stream from the final condenser reflux and are sent to the heads concentrating column (or the heads concentrating section in a modified Barbet beer still).

The control system used in the rectifier is usually the same as that used in the beer still concentrator, working on ?mid column? temperature.

Demethylation

When grain is used as the feedstock, the rectifier product is in fact the final product. However, where the feedstocks tend to produce more methanol as in the case of potatoes, grapes, and to some extent molasses, the rectifier product is sent to a demethylizing column. This column may have about 50 trays and will have indirect heating via a reboiler at the base.

The rectifier product is fed into the demethylizer at about tray 25 or 30. The methanol and any other heads components rise up through the column and concentrate at the top. They are drawn off as a heads purge stream and either sent to the heads concentrating column or sold as low grade industrial alcohol. The ethanol and any higher alcohol congeners in the rectifier product will descend the column and emerge from the base as the final product.

Variations in rectification systems

It is not possible to cover in this chapter all the variations in rectification systems used for neutral spirit production. However, one common variation is a fusel oil concentrating column which receives the fusel flows from the beer still concentrator and from the rectifier. The isoamyl alcohol concentrates in this column and is then sent to the fusel oil decanter for separation.

However, if the proofs are controlled steadily in the other columns to concentrate the isoamyl alcohol sufficiently for a direct feed to the decanter, this column should not be necessary.

There are several variations in column arrangements that may save energy but do not materially affect overall neutral spirit production.

For example, the extractive distillation column may be operated under pressure to heat the demethylizer. (In effect, the reboiler on the demethylizer becomes the first condenser for the extractive distillation column). Similarly, the rectifier may be operated under pressure to ?cascade? heat to a reboiler on the beer still, which in turn may be operated under vacuum to save energy and reduce the incidence of scaling.

Integration of beer still and rectification systems

Figure 7 shows an integrated beer still and rectifying system for the production of neutral spirit. In this example, a modified Barbet beer still is shown with the heads from the concentrator and the rectifier being

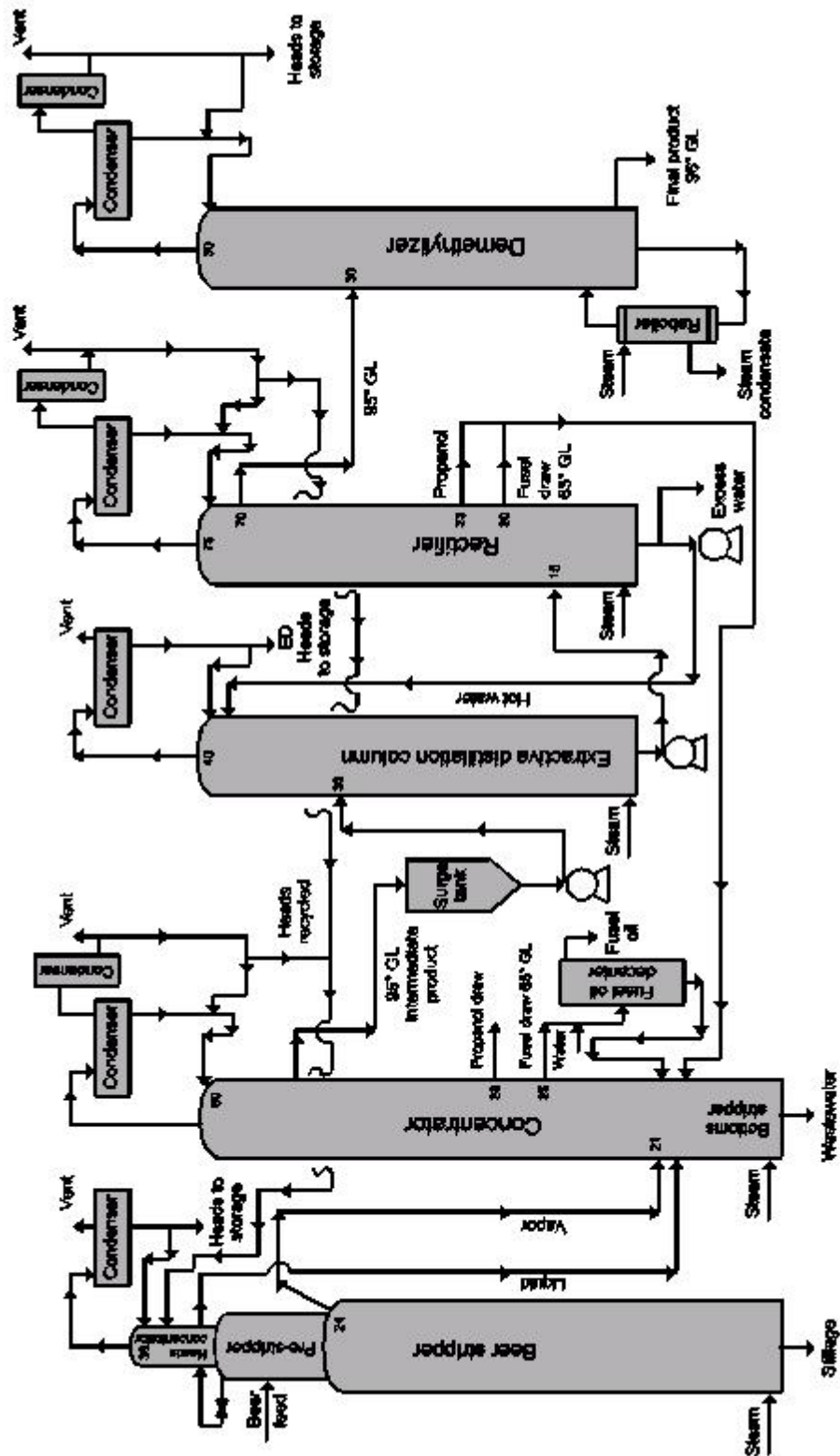


Figure 7. Complete beer still and rectification system used in neutral spirit production.

concentrated in the heads concentrator located on top of the beer stripper.

Handling and storage of neutral spirit

As neutral spirit should be odorless and tasteless,

care must be taken in handling and storage to prevent it acquiring odors and tastes from other materials. Any piping and tanks used for neutral spirit should be checked carefully, drained and flushed with some neutral spirit (which is recycled to distillation) prior to use. Contrary to usual beliefs, storage tanks need not be fabricated from stainless steel. Carbon steel tanks are quite acceptable provided that the main discharge valve is located above the bottom of the tank to allow rust accumulating in the sump to be drained. Rubber or plastic hoses should be avoided if possible, and flexible stainless steel hoses used where necessary. If rubber or plastic hoses or gaskets must be used, they should first be tested for any reaction with ethanol. This may be done by immersing shavings of the material in a jar of neutral spirit for at least 48 hrs. Compare a sample of the spirit diluted to about 20

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visually and organoleptically with a fresh sample of the same neutral spirit. If there is some cloudiness (due to hydrocarbons), or if the sample has picked up an odor not present in the original, the hose or gasket is unacceptable.

Process control

The process control used in neutral spirit production is much the same as that used in the production of whisky, rum or fuel ethanol, but in general more use is made of gas chromatography and organoleptic testing. The final product should be run into daily production tanks (closed receivers). These tanks should be switched periodically (normally every 24 hrs) and isolated until the product has been approved. The normal tests conducted are proof, acidity, gas chromatography and organoleptic evaluation.

There are no official standards for neutral spirit in the US; so specifications are established between the supplier and purchaser. In 1983, the European Community established the specifications for neutral spirit (Table 1). These specifications included an extraordinarily high limit of 500 ppm for methanol. This political decision appeased Northern European ethanol producers who use potatoes and grapes as feedstocks. Their product consequently had high levels of methanol; and they did not want to be forced to invest in demethylizers. In practice, however, European ethanol purchasers generally set a maximum specification of 50 ppm for methanol. The standards for neutral spirit in Mexico, Bolivia, Brazil and Ecuador are also shown in Table 1. It will be noted that the standards vary greatly and that the Ecuadorian standard, which is one of the most stringent, also includes a limit of 45 ppm for all congeners.

Gas chromatographic analyses should not be relied upon exclusively in grading neutral spirit. Organoleptic testing should be considered at least equally important as there are many compounds such as diacetyl with odor thresholds well below levels detected by normal gas chromatography.

Very often plants have two quality grades for neutral spirit. The top quality is for use in the preparation of vodka, while the second is sold to less discriminating customers who can accept a trace of odor or taste or a slight excess of a congener in the gas chromatographic analysis.

Any product failing to meet these grades is either sold as a mid-grade industrial alcohol or is reprocessed through the rectification system.

In controlling the process, it is advisable to take samples periodically from all of the draw points on the columns (the fusel oil, other higher alcohols, heads, and intermediate product valves). Additionally, use gas chromatography and sniffing to detect excessive accumulation of any congeners and adjust the purge draw rates accordingly. Some distillers recommend daily checking of the propanol concentration in the higher alcohol draws as high levels at these points could lead to 'spillovers' into the beer still or rectifier products.

Table 1. Specifications for neutral alcohol.

*expressed in mg per 100 mls alcohol at 100% volume.

Organoleptic testing is usually performed on samples of neutral spirit that have been diluted with odorless water to about 20

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various methods of comparison, but a preferred method is to have one sample glass marked as the standard and another standard sample identified only by a number or letter included with four or five other samples to be checked, which are similarly lettered or numbered. The persons performing the test should be unaware of the identities of the samples or the hidden standard until after completion. They should rate the samples on a 1-10 scale similar to that below:

10Excellent - no discernible odor
9Very good - only a very slight odor
8Good - no unpleasant odor
7Slightly good - slight unpleasant odor
6Borderline - noticeable unpleasant odor
5Unacceptable - yoo much off odor
4Bad
3Very bad
2Terrible - unpleasant
1Sickening

If a product quality problem cannot be corrected easily by increasing the draw rate of heads or other congeners, the best action is to slow the rectification operation and reduce throughput until the problem is corrected. In extreme cases, the rectifier may be put on ?total reflux?. This is a situation where the feed rate to the extractive distillation column is reduced to the point where there is just enough volume to meet the requirements of the congener draws, and none or almost no excess for the product draw. Under these conditions, quality problems can be corrected rapidly and the extractive distillation column feed rate can then be gradually increased to normal maximum rate.

It is obvious that obtaining a good ?clean? sample to use as an organoleptic standard is of great importance. Initially the standard may be obtained from another distillery, but a good opportunity to obtain a sample for use as a standard is at the end of an extended period of total reflux.

Production of vodka

The 1982 US Bureau of Alcohol, Tobacco and Firearms (BATF) regulations define vodka as ?neutral spirit so distilled or so treated after distillation, with charcoal or other materials, as to be without distinctive character, aroma, taste or color?. The use of charcoal filtration is now optional, whereas in earlier regulations it was mandatory and even the time and amounts of fresh charcoal to be used were specified. This change is in recognition of the relatively recent improvements in the quality of neutral spirits. Vodka is generally taken to be odorless, tasteless and colorless ethanol, but in the past in Eastern Europe vodka was lightly flavored with grasses or herbal extracts. It should be stressed that not only is the charcoal treatment nonessential, it is also not particularly effective and will not make a poor quality, improperly rectified neutral spirit into a good quality vodka.

Neutral spirit should be diluted to about 55

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before charcoal filtration. The old BATF regulations (1961) specified dilution to between 55

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and 40

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GL at a minimum contact time of 8 hrs with 10% of the charcoal replaced every 40 hrs to give a minimum usage of 6 lbs of new charcoal per 100 gallons of spirit treated. This was usually achieved by passing the diluted spirit through a series of eight or nine cylindrical charcoal filtration beds in a slow, continuous flow with one of the beds changed every day. The fresh bed would be connected last in the series. This meant that the beds were constantly being rotated; so the preferred arrangement was to set the beds in a circle to facilitate the changing. A simple alternative treatment method is to add charcoal to diluted neutral spirit in a tank and agitate or circulate it through a pump for a suitable length of time.

The water used in the initial and final dilutions should be clean, odorless and preferably demineralized. The demineralization is generally for aesthetic purposes as consumers do not like to see a white film of salts around the side of a bottle or glass if the vodka has been allowed to evaporate.

In countries where laws require that all ?spirits? be aged in wooden barrels, it may be necessary to add a small amount of sugar and/or glycerine to be able to classify vodka as a liqueur or ?compound spirit? rather than as an ?immature spirit?. The amount of sugar or glycerine used is normally the minimum required to provide ?proof obscuration?. This occurs when there is sufficient dissolved material to cause the apparent proof obtained by direct testing to differ fractionally from the real proof obtained by distilling the ethanol from a sample in a laboratory still and retesting after redilution to the original sample volume.

Great care should be taken in the bottling of vodka to prevent contamination with residues of other odorous products. The tanks and bottling systems should be washed thoroughly if previously used for other products. However, some bottlers prefer to keep a set of tanks and a

bottling line dedicated solely to handling vodka.
For further reading on vodka processing see
the reviews by Simpson (1977)

and Clutton
(1979).

Production of gin

The BATF definition of gin is
?a product obtained by original distillation
from mash, or by redistillation of distilled
spirits, or by mixing neutral spirits with or
over juniper berries and other aromatics,
or with or over other extracts derived from
infusions, percolations, or maceration of
such materials, and includes mixtures of gin
and neutral spirits. It shall derive its main
characteristic flavor from juniper berries
and be bottled at not less than 80

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proof
(40

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GL). Gin produced exclusively by
original distillation or redistillation may be
further designated as ?distilled?.
The regulation also states that dry gin (London
dry gin), Geneva gin (Hollands gin) and Old Tom
gin (Tom gin) are types of gin known under such
designations.
This regulation means that gin may be
produced by 1) distilling spirit with juniper berries
and other botanicals, or 2) mixing spirit with a
distilled gin concentrate, or 3) mixing spirit with
a blend of essences of juniper and other
flavorings.
The spirit used in gin production is usually
neutral, but in the production of Geneva gin,
which is popular in the Netherlands and Quebec,
it is a heavily flavored distillate referred to as ?malt
wine?.
?Distilled gin? is normally produced in batch
operations using pot stills. The pot still is usually
filled with neutral spirit diluted to 45-60

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then the juniper berries and other botanicals are
added. The berries and botanicals may be added
directly to the spirit either in loose form or
contained in a cotton sack. Alternatively, the
mixed botanicals may be suspended above the
liquid surface either in a cotton sack or in a wire
mesh rack.
In the gin distilling process the pot still is
heated by steam indirectly through a calandria
in the bottom of the pot. The distillate coming
over in the first few minutes of flow is normally
discarded as heads for reprocessing. The main
bulk of the distillate is then taken as product, and
the final portion distilling below a predetermined
proof (of about 45
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GL) is discarded as tails for
reprocessing. The pot still product is then sent
to the bottling department for dilution and
bottling. There is usually no storage or blending
of different gin batches.
In the preparation of ?gin concentrate? the
distillation process is much the same as for
distilled gin, but a much greater quantity of
botanicals is added in the pot still. The gin
concentrate is then simply blended with neutral
spirit prior to bottling.
Gin essences are prepared by blending
essential oils and other extracts derived from
juniper berries and botanicals. With the
introduction of highly concentrated gin
essences, it is possible to use as little as 0.01%
by volume of the essence in a blend with neutral
spirit.
Some internationally known brands of gin are
produced by all three methods (i.e. distilling,

blending, and essence blending) in
different countries without appreciable variance
in taste and odor when normal quality control
procedures are used.
The quality and type of juniper berries and
the mix of other botanicals largely determines
the nature of the end product. For example, the
flavor of London dry gin is strongly influenced
by large amounts of coriander seeds in the
botanical mix. Simpson (1966; 1977) and Clutton
(1979) have listed several botanicals commonly
used in gin production (Table 2). Another
frequently used botanical is the chamomile
flower (*Chamaemelum nobile*).

Table 2. Botanicals used in production of gin.

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Common name
Botanical name
Juniper berries

Juniperis communis
 Coriander seed
 Coriandrum sativum
 Liquorice root
 Glycyrrhiza spp.
 Fennel seed
 Foeniculum vulgare
 Cubeb berries
 Piper cubeb
 Cinnamon bark
 Cinnamomum zeylanicum
 Nutmeg
 Myristica fragrans
 Aniseed
 Pimpinella anisum
 Grains of paradise
 Afromomum melegueta
 Cassia bark
 Cinnamomum cassia
 Sweet orange peel
 Citrus sinensis
 Bitter orange peel
 Citrus aurantium
 Cardamom seeds
 Elettaria cardamomum
 Angelica root
 Archangelica officinalis
 Lemon peel
 Citrus limon
 Orris root
 Iris pallida
 Callamus root
 Acorus calamus
 Caraway seed
 Corum carvi

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Adapted from Simpson (1966, 1977) and Clutton (1979).

As with vodka, great care should be taken in handling and bottling gin. Unlike vodka,

however, the problem is not picking up flavors from other products. The risk is contamination of other products with gin. If it is not possible to use a dedicated set of tanks and bottling equipment, everything coming in contact with gin should be thoroughly washed before use on any other beverage.

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